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Influence of Column Support on Separation of Fatty Acid Methyl Esters by Gas Chromatography

SIR: We reported the use of a poly (vinyl acetate) (PVA) liquid phase on Chromosorb, 30–60 mesh, for the separation of fatty acid methyl esters (*4*). Subsequently, batches of Chromosorb R and W (both diatomaceous earth products) when used as supports for PVA gave separations of widely varying efficiencies. Horning, Moscatelli, and Sweeley (*3*), following the procedure of Howard and Martin (*5*), reported that screened Celite 545, 60–80 and 80–100 mesh, acid-washed, dried, treated with vapors of dimethyldichlorosilane, and then washed with methanol, gave a superior column support for polyester-type liquid phases. More recently, Hishta *et al.* (*1*, *2*) suggested the use of microbeads as the solid support for liquid substrates, in the order of 0.25% by weight, to separate high boiling compounds at a rapid rate and at relatively low column temperatures.

A comparison of the separations obtained for several fatty acid methyl esters on PVA, supported on "silanized" 30–60 mesh Chromosorb R and Celite 545 and on waterproofed glass microbeads, 30–60 mesh, is given in Table I. Although the Celite 545 fractions can be pretreated satisfactorily by passing nitrogen saturated with dimethyldichlorosilane through a 100-gram batch of the support for approximately one hour, Chromosorb R required a very much longer exposure time to the vapors of dimethyldichlorosilane. Best results in the latter case were obtained by exposing the granules to dimethyldichlorosilane vapors in a closed container for 2 weeks and then washing with methanol.

Fatty acid ester analyses were made, using 9-foot, coiled copper columns, 1/4-inch outside diameter, 0.03-inch wall thickness. The partitioning medium was 15% PVA by weight on Chromosorb R and Celite 545, respectively, and 0.25% PVA by weight on the glass microbeads. The helium flow rate for all experiments was adjusted to 83 ml. per minute measured at the column exit. The detector was of the thermal conductivity type, and the samples analyzed contained approximately 300 μg. of the mixed esters. The temperature of the column and detector was 205° C. for the Chromosorb R and Celite 545 columns. For the glass bead columns, several temperatures were used; results are reported for an operating temperature of 168° C. In Table I, the separation factors are the ratios of retention volumes compared to the retention volume of the C_{17} margaric acid. The theoretical plates were calculated in conventional manner by dividing the distance from the "air pip" to the peak in question, by the peak width at the base, squaring this quantity, and multiplying by 16.

Results, using treated Chromosorb R and Celite 545, were uniformly good. The close check of the separation factors on these two PVA coated supports indicates that this treatment produced a reproducible, uniform, and similar surface on both materials. Microscopic observation of the coated granules showed marked difference in their surfaces, the granules of Celite 545 being more jagged in appearance than those of Chromosorb R. It may be that the differences in physical structure between these two materials are responsible for the difference in observed retention volumes. The use of glass microbeads as a support gave rapid results at relatively low operating temperature; however, the peaks were too poorly resolved, under our operating conditions, to make this column useful. If the glass microbead column is similar in performance to a capillary-type column, the poor resolution may be attributed to overloading. The glass microbead column may, therefore, be useful with ionization-type detectors, where smaller samples can be used, but unsuitable for use with thermal conductivity detectors.

LITERATURE CITED

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Table I. Separation Factors, Retention Volumes, and Theoretical Plates (9-Foot Column) for Fatty Acid Methyl Esters on Poly(vinyl Acetate)

	Column Support								
	Chromosorb R, T = 205° C.			Celite 545, T = 205° C.			Glass Microbeads, T = 168° C.		
Methyl Ester	Retention volume, ml.	Separation factor	Theoretical plates	Retention volume, ml.	Separation factor	Theoretical plates	Retention volume, ml.	Separation factor	Theoretical plates
Laurate	452	0.23	1190	248	0.23	640	85	0.16	16
Myristate	798	0.41	1600	444	0.41	910	177	0.33	32
Palmitate	1438	0.73	1760	803	0.73	950	366	0.69	34
Margarate	1962	1.00	1820	1097	1.00	1470	530	1.00	73
Stearate	2580	1.32	1925	1450	1.32	1500	750	1.41	94
Oleate	2890	1.47	2050	1633	1.48	1735	850	1.60	139
Linoleate	3370	1.72	2120	1913	1.75	2035	...	...	...